ANALYST:		VPDES NO	

Instrument: _____ Parameter: Mercury by Cold Vapor

Checklist

04/02

METHOD OF ANALYSIS

	EPA 245.1-2 Revision 3.0
	18th Edition of Standard Methods 3112B

		Y	N
	SAMPLE COLLECTION AND PRESERVATION		
1)	Are all glassware and plasticware acid washed? (Detergent and tap water wash with following rinses: 1:1 HNO_3, tap water, 1:1 HCl, tap water, and final DI water rinse.) If not, were blanks run to ensure acceptability of procedure and materials? [EPA-6.5; SM-3010 C.2]		
2)	Are samples for total mercury (Hg) unfiltered and preserved with HNO_3 to a pH of <2? [EPA-8.2; SM-3030 A]		
3)	Are samples for dissolved Hg filtered through a 0.45µ membrane filter immediately after collection and preserved immediately after filtration with HNO_3 to a pH of <2? [40 CFR, Part 136.3 Table II, footnote 7; SM-3030 B]		
4)	For dissolved Hg, were the filtration apparatus and filter rinsed with 50-100 mLs of sample/water and the filtrate discarded prior to filtering the aliquot to be preserved as the dissolved Hg sample? [SM3030 B]		
	EQUIPMENT		
5)	Is either an AA spectrometer with a Hg lamp or a commercial mercury system used for analysis? [EPA-6.1.2; SM3112 B.2.a]		
6)	Are instrument responses electronically recorded either by strip chart or computer? [EPA-6.1.7; SM3111 A.6.c]		
7)	Is instrument allowed an appropriate warm up time? [EPA-10.1; SM3112 B.4.a]		
8)	Is the absorption cell of approximately 1" OD by 4.5" long with quartz windows cemented to both ends? [EPA-6.1.3; SM3112 B.2.b]		
9)	Is the aeration tube used in sample bottles made of straight glass ending with frit of coarse porosity? [EPA-6.1.4; SM3112 B.2.f]		
10)	If micropipettes are used, are the critical volumes checked quarterly for accuracy and precision? [Permit]		
	SAMPLE PREP		
11)	Are all glassware and plasticware acid washed with detergent and tap water, rinsed with 1:1 HNO_3, tap water, 1:1 HCl, tap water, and final DI water rinse? If not, were blanks run to ensure acceptability of procedure and materials? [EPA-6.5; SM-3010 C.2]		
12)	Is the area where Hg samples are prepared and analyzed free from dust and other sources of possible Hg contamination, such as noncompatible analyses (COD and TKN) and uncovered fluorescent light bulbs? [EPA-4.3; SM-3112 B.2]		
13)	Are all reagents of low Hg content (may need trace metal grade)? Solid reagents should not exceed 0.05 ppm of Hg. [EPA-7.1]		
14)	Are reagents being dated and initialed on the container upon receipt and when opened? [Permit]		
15)	Are reagent and standard solutions that have been prepared on site, dated and initialed by the analyst? NOTE: It is recommended that a log be maintained with the lot numbers of the chemicals used in each preparation. [Permit]		

Mercury

		Y	N
16)	Are digestion fumes removed by a fume hood or evacuation equipment? [EPA-5.2; Permit]		
17)	Is a digestion log being maintained with the following information recorded? [Permit]		
	a) Date, time, method, and analyst performing digestion.		
	b) Sample ID number with beginning volume.		
	c) pH of sample taken just prior to digestion. NOTE: If pH is > 2, add HNO_3 and hold sample for 16 hrs. until verified to be pH < 2. [EPA-8.2]		
	d) Reagents and volume of each one used.		
	e) ID of digestion container used.		
	f) Samples used for quality control are properly identified [blanks, laboratory control samples (LCS), spikes, and duplicates]		
	g) Conc., volume, and source of spiking solution used.		
18)	Are stock standards within expiration dates? [Permit]		
19)	Is Hg standard prepared fresh each day of use (v/v= 0.15% HNO_3)? [Permit; SM3112 B.3.c]		
20)	Are calibration standards and check samples prepared by transferring aliquots of the working solution to Hg bottles and diluting to a final volume of 100 mLs? [EPA-11.1.1; SM-3112 B.4.b]		
21)	Are 100 mLs of sample, or an aliquot diluted to 100 mLs, transferred to Hg bottles? [EPA-11.1.1; SM-3112 B.4.c]		
22)	Are the following digestion reagents added to standards, blanks, samples, and quality control samples? [EPA-11.1; SM-3112 B.4.b/c]		
	a) 5 mL conc H_2SO_4		
	b) 2.5 mL conc HNO_3		
	c) 15 mLs potassium permanganate ($KMnO_4$). If necessary, add additional amounts of $KMnO_4$ until a purple color persists for 15 min.		
	d) 8 mL potassium persulfate ($K_2S_2O_2$)		
23)	Are samples heated in a covered hot water bath maintained at 95°C for 2 hrs? (NOTE: Timing should begin when the temp reaches 95°C.) [EPA-11.1.3; SM-3112 B.4.b]		
24)	Are samples allowed to reach room temperature prior to beginning analysis? [EPA-11.1.4; SM-3112 B.4.b]		
25)	Is ≥6 mL of hydroxylamine solution added per bottle to reduce excess permanganate? [EPA-11.1.5; SM-3112 B.4.b]		
	ANALYSIS		
26)	Has initial demonstration of performance been completed? [EPA-9.2; SM1020 B.1]		
	a) Has a quality control sample been analyzed with recovery ± 10% of stated value? [EPA-9.2.3; SM-3112 B.4.b]		
	b) Has the method detection limit (MDL) been determined? Must be done annually. [EPA-9.2.4; SM1020 B.1]		
27)	Are Hg bottle solutions aerated after addition of hydroxylamine solution? [SM-3112 B.4.c]		
28)	Are 5 mL of stannous solution added and immediately attached to the aeration tube? [EPA-11.2.3; SM-3112 B.4.b] For automated systems, are manufacturer's instructions followed?		

Mercury

		Y	N
29)	Is the absorbance allowed to reach a maximum height prior to taking a reading? [EPA-11.2.3; SM-3112 B.4.b]		
30)	Is the system purged and the recorder allowed to return to minimum value prior to reading the next bottle? [EPA-11.2.3; SM-3112 B.4.b]		
31)	Are Hg vapors vented or passed through a trapping solution or through activated charcoal? [EPA-6.1.6; SM-3112 B.2.h]		
32)	Does the calibration curve consist of a blank and at least 5 standards for EPA, 3 for SM, and have a calibration coefficient of ≥0.995? [EPA-11.2.2; SM-3112 B.4.b]		
33)	Is an instrument performance check (made from same source as standards) and calibration blank analyzed immediately after calibration (ICV), after every 10th sample (CCV), and at end of sample run (CCV)? Recoveries must be within ± 5% for ICV and ± 10% for CCV. [EPA-9.3.4; SM-3020]		
34)	Is a quality control sample(QCS) made from outside source different from standards, analyzed to verify each calibration? Recovery within ± 10%. [EPA-9.3.2; SM-3020]		
35)	Is a laboratory fortified blank (LFB) analyzed with each batch of samples with a spike recovery of ± 15%? [EPA-9.3.2; SM-3020]		
36)	Are a minimum of 10% of samples being spiked prior to digestion? [EPA-9.4.2; SM-3020]		
	a) Is recovery within 70-130% [EPA-9.4.3; 85-115% [SM3111 A.7]?		
	b) If recovery is outside the acceptable range, and the laboratory performance is shown to be in control, are the results flagged as being suspect due to matrix effects? [EPA-9.4.4]		
37)	Are duplicate samples being analyzed at rate of 20% with RPD < 20%? (RPD's are valid only for values > 5X the CRDL.) [EPA-9.4.1; SM3020]		
38)	Are reported sample values bracketed by standards? (Values below the lowest standard must be reported as <.) [Permit]		
	DATA KEEPING		
39)	Does raw data have analyst's initials, analysis time and date recorded? [Permit]		
40)	Are all raw data retained for at least 3 years? [Permit]		
41)	Is there a QA/QC plan available which includes a 'Corrective Actions' section? [Permit]		

PROBLEMS:

Mercury